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Gas Chromatographic/Fourier Transform Infrared Spectrometric Identification of Hazardous Waste Extract Components

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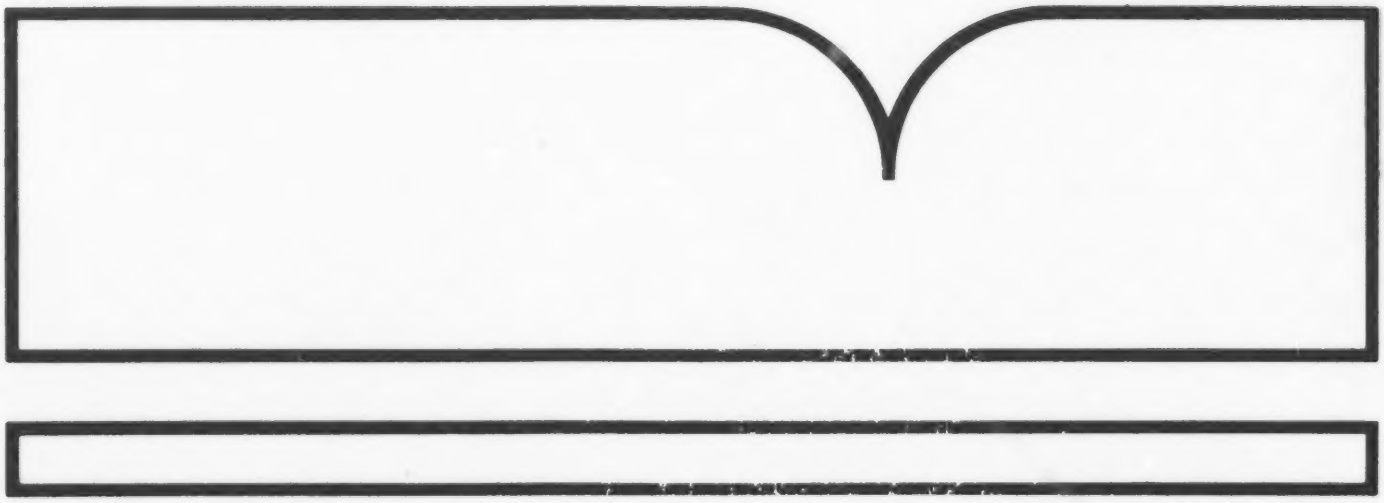

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16. ABSTRACT

The Resource Conservation and Recovery Act of 1976 (RCRA) and its amendments require the U.S. Environmental Protection Agency to institute a program to control hazardous waste. Implementation of the RCRA will require reliable monitoring tools for the characterization of a broad spectrum of hazardous wastes. Accordingly, the capability of packed column gas chromatography/Fourier transform infrared spectrometry (GC/FT-IR) using the Gas Infrared Fourier Transform Software (GIFTS) to analyze complex hazardous waste extracts was tested. These extracts were also analyzed by gas chromatography/mass spectrometry (GC/MS) and the on-line library search results for both techniques were compared. GC/FT-IR was a powerful tool for aromatic isomer differentiation in these complex wastes. The GIFTS on-line library search correctly identified poorly resolved gas chromatographic effluent components. The magnitude of the GIFTS hit index difference between the first and second library search hits is a reliable indicator of the search validity. The combination of GC/FT-IR and GC/MS may provide the identification of all volatile components in complex environmental extracts.

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Gas Chromatographic/Fourier Transform Infrared Spectrometric Identification of Hazardous Waste Extract Components

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The Resource Conservation and Recovery Act of 1976 (RCRA) and its amendments require the U.S. Environmental Protection Agency to institute a program to control hazardous waste. Implementation of the RCRA will require reliable monitoring tools for the characterization of a broad spectrum of hazardous wastes. Accordingly, the capability of packed column gas chromatography/Fourier transform infrared spectrometry (GC/FT-IR) using the Gas Infrared Fourier Transform Software (GIFTS) to analyze complex hazardous waste extracts was tested. These extracts were also analyzed by gas chromatography/mass spectrometry (GC/MS) and the on-line library search results for both techniques were compared. GC/FT-IR was a powerful tool for aromatic isomer differentiation in these complex wastes. The GIFTS on-line library search correctly identified poorly resolved gas chromatographic effluent components. The magnitude of the GIFTS hit index difference between the first and second library search hits is a reliable indicator of the search validity. The combination of GC/FT-IR and GC/MS may provide the identification of all volatile components in complex environmental extracts.

The Resource Conservation and Recovery Act of 1976 (RCRA) and its amendments require the U.S. Environmental Protection Agency (EPA) to regulate hazardous waste activities (*1*). Under the provisions of RCRA amendments, criteria for hazardous waste classification include ignitability, corrosivity, reactivity, radioactivity, infectiousness, toxicity, phytotoxicity, teratogenicity, and mutagenicity (*2*). Implementation and enforcement of the RCRA require reliable monitoring tools. These tools must be capable of detecting and identifying hazardous substances in complex mixtures at concentrations ranging from pure compounds to dilute solutions.

Gas chromatography/mass spectrometry (GC/MS) has been the tool most commonly employed by the EPA for the unequivocal identification of volatile and semivolatile hazardous substances (*3*, *4*). However, GC/MS is only one of an array of analytical tools, and the unequivocal identification of organic chemicals may best be accomplished by utilizing several spectral techniques in a complementary fashion (*5–7*). Available spectral techniques for organic analysis include infrared (IR), mass (MS), nuclear magnetic resonance (NMR), ultraviolet–visible (UV), and fluorescence spectrometries. UV and fluorescence spectrometries only provide information about the electronic distribution within molecules. Proton NMR identification limits are currently in the microgram range with analysis times of several minutes per compound (*8*, *9*). In addition the technique of routine NMR continuous flow analysis has not yet been perfected (*9*, *10*).

The emergence of gas chromatography/Fourier transform infrared (GC/FT-IR) techniques has provided a second spectral method of sufficient sensitivity and rapidity for on-line environmental analysis (for a recent review of the GC/FT-IR method see ref 11). Preliminary reports of the GC/FT-IR method suggest a promising role for this technique in "real world" sample analysis (*6*, *12*, *13*). The recent development of powerful search algorithms with the capability to perform on-line library searches of vapor-phase infrared spectra should greatly increase the utility of the GC/FT-IR method (*14–18*). This report demonstrates the application of one of these new algorithms (GIFTS) to the detailed analysis of complex hazardous waste extracts.

EXPERIMENTAL SECTION

Extract Preparation. Extracts of waste-contaminated soil samples were prepared in the following manner: To a 2-L round-bottom flask on a magnetic stirring apparatus add 50 g of soil. Then add 800 mL of distilled water and stir 1 min. Acidify with 8 mL of concentrated nitric acid and stir an additional 10 min. Attach the round-bottom flask to a Neilsen-Kryger steam distillation apparatus containing 5 mL of distilled water and 15 mL of nanograde hexane in the condenser solvent reservoir. Stir and reflux for 1 h. Separate the hexane layer and wash the solvent layer with an additional 50 mL of hexane. Pour the combined hexane extracts through a drying column of organics-free anhydrous sodium sulfate. Add the dried hexane extract to a 500-mL Kuderna-Danish flask fitted with a 10-mL concentrator tube. Attach a three-ball macro-Snyder column and condense the extract to about 4 mL in a 65 °C water bath. Replace the macro-Snyder column with a micro-Snyder column and reduce the extract volume to 1 mL using a stream of organics-free nitrogen.

The chemical still bottom extract was prepared by a previously described procedure (*19*).

GC/FT-IR Instrumentation. All GC/FT-IR experiments were performed on a Digilab FTS-20B spectrometer equipped with a 15-bit A/D convertor and a narrow-band, 2-mm^2 focal chip, mercury–cadmium telluride (MCT) detector. GC/FT-IR separations were performed on a Hewlett-Packard Model 5880A gas chromatograph. The GC column was 6 ft × $^1/_8$ in. i.d. and packed with 1.5% OV-17 and 1.95% QF-1 on 80/100 mesh Gas-Chrom Q. Helium flow was 30 mL/min and the gas chromatograph was programmed from 70 to 225 °C at 10 deg/min. Details of this GC/FT-IR system and its operation have been described elsewhere (*20*).

GIFTS Software. This software has been described elsewhere (*14–16*). GIFTS revision 2.0 containing a 2300 vapor-phase spectral search library was purchased from Digilab Inc. and utilized in this study. The 2300 vapor-phase spectra and accompanying software were supplied by Kenneth Shafer of the Battelle—Columbus Laboratories. The on-line library search identification of every extract component was verified by visual comparison with standard vapor-phase infrared spectra. Every spectrum was referenced against 20 scans of the empty light pipe.

GC/MS Instrumentation. GC/MS experiments were performed on a Finnigan Model 4023 quadrupole mass spectrometer. The mass spectrometer was operated at the following conditions: electron energy, 70 eV; electron multiplier, 1325 V; and source temperature, 240 °C. Masses 45–450 were scanned in 1 s. Data were collected and processed on a Finnigan/Incos data system with a Nova 3 computer. The spectrometer was mass calibrated with perfluorotri-*n*-butylamine.

GC/MS Operation. GC/MS separations were performed on a Finnigan Model 9610 gas chromatograph equipped with a Grob type split/splitless injector. A 0.32 mm i.d. × 30 mm SE-54 fused silica capillary column was coupled directly to the source. Splitless injection was used and after a 4-min hold at 35 °C the GC oven was ramped to 265 °C at 10 deg/min.

RESULTS AND DISCUSSION

Selection of Sample Extracts. Two soil extracts from abandoned chemical dump sites and the extract from an industrial chemical still were analyzed by gas chromatography/mass spectrometry (GC/MS). Each of these extracts contained over 50 GC/MS detectable components. The GC/MS on-line library search indicated that the soil extracts contained many isomeric chlorinated benzenes and toluenes. The GC/MS electron impact spectra of these isomeric aromatic compounds were similar (see Figure 1) and GC/MS identification could only be achieved by using the GC retention times of standard compounds. Since the ability of infrared spectroscopy to differentiate among isomeric aromatic compounds is well established (see Figure 2) and the capability of GC/FT-IR to analyze hazardous waste extracts had previously been demonstrated (*20–23*), these extracts were reanalyzed by GC/FT-IR.

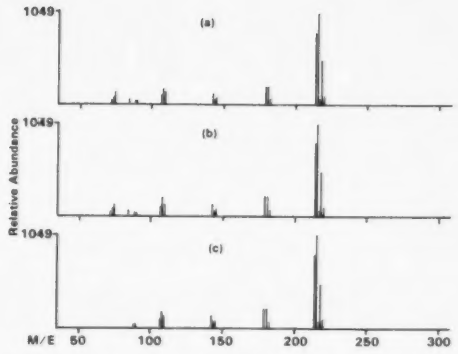

Figure 1. Electron impact mass spectra of the isomeric tetrachlorobenzenes: (a) 1,2,3,5-tetrachlorobenzene; (b) 1,2,3,4-tetrachlorobenzene; (c) 1,2,4,5-tetrachlorobenzene.

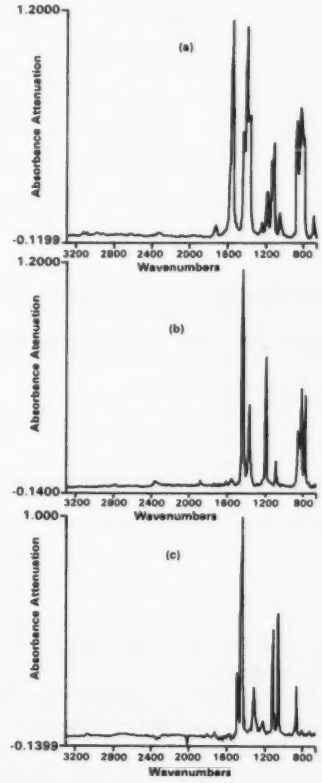

Figure 2. Vapor-phase infrared spectra of the isomeric tetrachlorobenzenes: (a) 1,2,3,5-tetrachlorobenzene; (b) 1,2,3,4-tetrachlorobenzene; (c) 1,2,4,5-tetrachlorobenzene.

GC/FT-IR and GC/MS Runs. The GC/FT-IR infrared reconstructed chromatogram (IRC) and GC/MS reconstructed ion chromatogram (RIC) of soil extract number two are shown in Figure 3. With the GIFTS software only a single GC/

Table I. GC/FT-IR and GC/MS On-Line Library Search Results for Soil Extracts and Chemical Still Bottom Extract

extract no.[a]	GC/FT-IR library search hit	GC/FT-IR scan no.	GC/FT-IR identification	GC/MS identification
3	1	56	methylene chloride	
3	1	83	trichloroethylene	trichloroethylene
2	1	119	toluene	
3, 2	1	131	tetrachloroethylene	tetrachloroethylene
3	1	176	1,1,1,2-tetrachloroethane[b]	1,1,1,2-tetrachloroethane
2		188	unidentified	
3	1	248	1,1,2,2-tetrachloroethane	1,1,2,2-tetrachloroethane
1, 2	1	272, 79	2-chlorotoluene	chlorotoluene
1, 2	1	280, 310	4-chlorotoluene	chlorotoluene
3	1	286	pentachloroethane	pentachloroethane
1, 2	1	342, 351	1,4-dichlorobenzene	dichlorobenzene
3	3	361	hexachloroethane	hexachloroethane
1, 2	1	364, 394	benzaldehyde	benzaldehyde
3		465	olefinic halide[b]	pentachlorobutadiene
1, 2	3	459, 478	2,4-dichlorotoluene	dichlorotoluene
2	2	480	2,5-dichlorotoluene	dichlorotoluene
1		496	3,4-dichlorotoluene	dichlorotoluene
3	1	498	hexachlorobutadiene	hexachlorobutadiene
2	1	547	1,2,4-trichlorobenzene	trichlorobenzene
2	1	566	4-chlorobenzaldehyde	chlorobenzaldehyde
1	1	572	1,2,3-trichlorobenzene	trichlorobenzene
2	1	595	4-chlorobenzyl chloride	dichlorotoluene
1, 2	1	646, 662	2,4,5-trichlorotoluene	trichlorotoluene
1, 2	1	687, 692	1,2,4,5-tetrachlorobenzene	tetrachlorobenzene
2	1	707	2,3,6-trichlorotoluene	trichlorotoluene
3	1	740	biphenyl	biphenyl
1, 2	1	755, 764	1,2,3,4-tetrachlorobenzene	tetrachlorobenzene
3, 1, 2	1	772, 776, 778	diphenyl ether	diphenyl ether
1	1	874	pentachlorobenzene	pentachlorobenzene
1	1	1034	benzyl ether	benzyl ether

[a] Extract numbers 1 and 2 are soil extracts. Extract number 3 is a chemical still bottom extract. [b] Not in vapor-phase infrared search library.

FT-IR run was necessary to collect and coadd the interferogram files, provide an infrared reconstructed chromatogram, transform the coadded files, record the IR spectra on magnetic disk, and perform the on-line library search. During these operations only a few minutes of user interaction, with the GC/FT-IR system, were required.

GC/FT-IR detected about one-third of the GC/MS detectable compounds in each extract. The GC/FT-IR and GC/MS compound identifications based on on-line library search results are shown in Table I. Each GC/FT-IR identification was confirmed by comparison of the extract component's IR spectrum with the vapor-phase IR spectrum of the library search hit. A total of 41 compounds were identified by GC/FT-IR in these three extracts, and visual comparison of the IR spectra with standard vapor-phase IR spectra indicated that 37 compounds were correctly identified on the first library search hit. The soil extracts were shown to contain the isomeric di-, tri-, and tetrachlorobenzenes. The mono-, di-, and trichlorotoluenes were also present. Identification was simplified since the search library contained all of the isomeric chlorobenzenes, monochlorotoluenes and dichlorotoluenes. However, only two of the six isomeric trichlorotoluenes are in this search library.

The industrial chemical still bottom analysis results in Table I (extract no. 3) indicate substantial agreement between GC/MS and GC/FT-IR on-line search results, provided the GC/FT-IR signal noise was sufficiently high for detection. GC/MS results suggest that the olefinic halide corresponding to GC/FT-IR scan file number 465 is pentachlorobutadiene, a compound type not contained in the vapor-phase infrared search library. This complementary on-line search agreement between two such dissimilar spectral techniques may eliminate the time, expense, and subjectivity of an operator comparison of unknown and standard spectra. Such an elimination is one of the most promising aspects of combined GC/MS, GC/FT-IR analysis.

GC/FT-IR Identification of Coeluting Extract Components. The infrared reconstructed chromatograms (IRC) of soil extracts one and two (Figure 3) suggested that many extract components were coeluting. It is conceivable that such coelution could lead to incorrect on-line library search results. This possibility was checked by comparing the IR spectra of the transformed interferogram files for each extract component with the standard vapor-phase IR spectra of the on-line library search hits. Examples of a few such spectral comparisons are shown in Figures 4 and 5. These figures depict the IR spectra of transformed scan files 547 and 566 of soil extract number two and standard vapor-phase IR spectra corresponding to the first library hit for each file. Transformed scan file 547 identified by the on-line library search as 1,2,4-trichlorobenzene is seen to contain a strong IR band at 1725 cm^{-1}, an impurity from the coeluting peak 566 which was identified as 4-chlorobenzaldehyde. Examination of the infrared spectra indicates that the trailing peak of the 547, 566 pair is pure.

Table II summarizes the on-line library search results for the three extracts. Of the 57 detected compounds 17 were shown to contain coeluting impurities by examination of their IR spectra. However, 12 of these 17 chromatographically impure compounds were correctly identified by the on-line library search.

Reliability of the GC/FT-IR Library Search. Since an environmental or hazardous waste extract could contain any of thousands of volatile organic compounds, any identification based solely on an on-line library search of a 2300 vapor-phase IR spectral library must be accepted with caution. In this study every GC/FT-IR identity assignment was made only after comparison of the IR spectra of unknowns with standard vapor-phase spectra. This is a time-consuming process so it

Table II. Summary of GC/FT-IR On-Line Library Search Results

extract	GC/FT-IR scan no.	GC/FT-IR identification	library hit index	hit index[a] difference
soil no. 1	272	2-chlorotoluene	0.22	0.27
	280[b]	4-chlorotoluene	0.33	0.25
	342	1,4-dichlorobenzene	0.28	0.28
	364	benzaldehyde	0.53	0.05
	459[b]	2,4-dichlorotoluene	0.37	0.16
	496[b]	3,4-dichlorotoluene	0.74	0.06
	533	1,2,4-trichlorobenzene	0.36	0.41
	572	1,2,3-trichlorobenzene	0.33	0.37
	587	alkane	0.63	0.03
	646	2,4,5-trichlorotoluene	0.52	0.28
	657	2,3,6-trichlorotoluene	0.42	0.41
	687[b]	1,2,4,5-tetrachlorobenzene	0.48	0.38
	764	1,2,3,4-tetrachlorobenzene	0.24	0.61
	778	diphenyl ether	0.58	0.03
	841	alkane	0.27	0.07
	874	pentachlorobenzene	0.59	0.45
	938	alkane	0.37	0.00
	1034	benzyl ether	0.41	0.06
soil no. 2	119	toluene	0.19	0.28
	128	tetrachloroethylene	0.30	0.44
	188	olefinic ether	0.70	0.00
	279	2-chlorotoluene	0.20	0.28
	310[b]	4-chlorotoluene	0.34	0.17
	351	1,4-dichlorobenzene	0.40	0.12
	394	benzaldehyde	0.37	0.10
	423[b]	unidentified	0.49	0.02
	478[b]	2,4-dichlorotoluene	0.44	0.00
	480[b]	2,5-dichlorotoluene	0.32	0.29
	495[b]	unidentified	0.28	0.03
	518[b]	unidentified	0.59	0.06
	547[b]	1,2,4-trichlorobenzene	0.64	0.13
	566	4-chlorobenzaldehyde	0.32	0.13
	586[b]	unidentified	0.84	0.01
	595	4-chlorobenzyl chloride	0.64	0.02
	603[b]	unidentified	0.69	0.00
	662[b]	2,4,5-trichlorotoluene	0.43	0.33
	692[b]	1,2,4,5-tetrachlorobenzene	0.52	0.33
	707	2,3,6-trichlorotoluene	0.71	0.11
	755[b]	1,2,3,4-tetrachlorobenzene	0.41	0.40
	772[b]	diphenyl ether	0.48	0.03
industrial still bottom	56	methylene chloride	0.30	0.31
	83	trichloroethylene	0.40	0.44
	131	tetrachloroethylene	0.35	0.34
	176	1,1,1,2-tetrachloroethane	0.52	0.07
	248	1,1,2,2-tetrachloroethane	0.46	0.03
	270	alkane	0.19	0.00
	286	pentachloroethane	0.26	0.35
	301	alkane	0.33	0.02
	361	hexachloroethane	0.74	0.05
	416	alkane	0.21	0.00
	465	olefinic halide	0.80	0.09
	498	hexachlorobutadiene	0.47	0.39
	566	alkane	0.36	0.00
	579	alkane	0.32	0.01
	685	alkyl halide	0.72	0.02
	740	biphenyl	0.32	0.06
	776	diphenyl ether	0.35	0.05

[a] Difference between library search hit indexes for first and second library hits. [b] Comparison of standard and extract component infrared spectra indicates coeluting impurity.

is of interest to examine the data derived from the on-line library search to ascertain the necessity for such operator comparisons. Table II summarizes the GC/FT-IR library searches for the three extracts. A total of 57 compounds were detected of which 41 were identified. Of the 16 unidentified compounds half were alkanes, a compound class difficult to identify by GC/FT-IR (*6*). Table II also includes the library hit indexes, a measure of the spectral fit of the unknown to the library hit (*15*). The arithmetic difference between the first and second hit indexes for each compound is also listed. The average hit index difference is 0.02 for the unidentified compounds and 0.23 for the 41 identified compounds. Seventy-one percent of the compounds correctly identified by the first on-line library search hit exhibited hit index differences greater than 0.10. This suggests that the magnitude of the GIFTS hit index difference between the first and second library hits is a reliable indicator of the on-line search validity.

Chromatographic Considerations. Although some SCOT (*24*) and WCOT (*25*, *26*) capillary column GC/FT-IR analytical results have been reported, these studies did not include the analysis of complex sample extracts. Currently, packed column GC/FT-IR systems are in greatest usage as a result

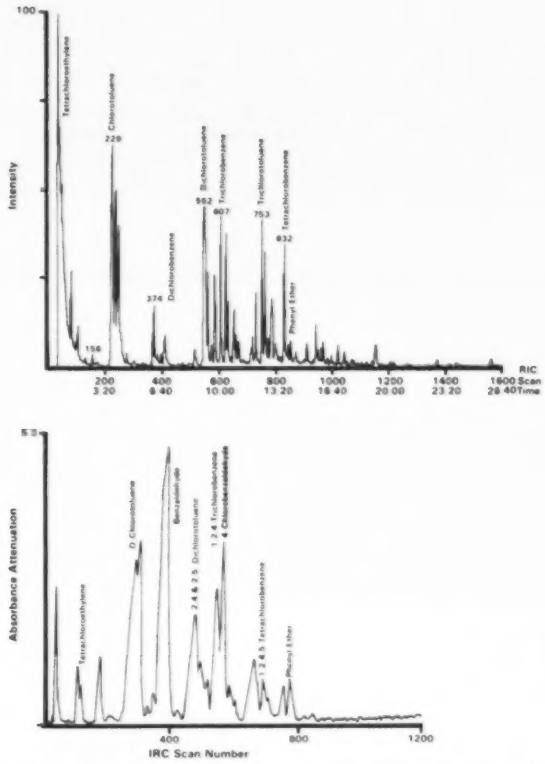

Figure 3. Total ion and infrared reconstructed chromatograms of soil extract number 2.

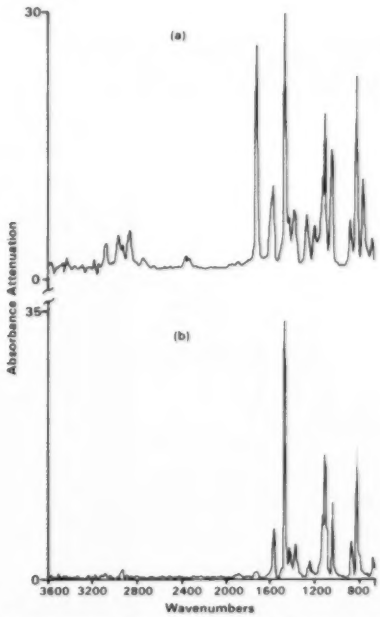

Figure 4. Soil extract number 2: (a) GC/FT-IR scan file number 547 identified as 1,2,4-trichlorobenzene; (b) standard vapor-phase infrared spectrum of 1,2,4-trichlorobenzene.

of their commercial availability but a capillary GC/FT-IR interface will soon enter the market (*27*).

The relative merits of a packed and a WCOT capillary column GC/FT-IR interface have recently been presented (*28*). The WCOT interface greatly reduced coelution problems while improving the signal noise (S/N) by a factor of 10. In addition the capillary GC/FT-IR allows a direct peak-by-peak

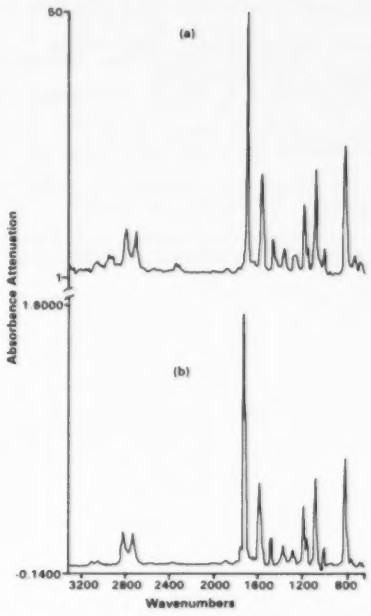

Figure 5. Soil extract number 2: (a) GC/FT-IR scan file number 566 identified as 4-chlorobenzaldehyde; (b) standard vapor-phase infrared spectrum of 4-chlorobenzaldehyde.

comparison of eluant peaks in the GC/MS and GC/FT-IR reconstructed chromatograms (*26*).

However, a recent paper by Rosenthal (*29*) suggests that these extracts which each contain more than 50 components, even if analyzed on a capillary column, will still contain at least five coeluting components.

Furthermore, since environmental extracts often contain components which span a wide concentration range, and since adjustment of extract volumes to prevent column overloading may dilute the lower concentration components below the GC/FT-IR detection limits, it is anticipated that packed column GC/FT-IR interfaces will still find use in environmental analysis.

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